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IS 1060-1 (1966): Methods of sampling and test for paper and allied products: Part 1 [CHD 15: Paper and its products]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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IS : 1060 (Part 1) – 1966
(Reaffirmed 2004)

Indian Standard

**METHODS OF SAMPLING AND
TEST FOR PAPER AND
ALLIED PRODUCTS : PART 1**

(Revised)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

METHODS OF SAMPLING AND TEST FOR PAPER AND ALLIED PRODUCTS: PART I

(Revised)

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(Continued on page 2)

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(Continued from page 1)

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13.2.2.2 Accessories

- a) Stop watch;
- b) Sheet of blotting paper of 250 g/m² and of 70 mm capillary rise;
- c) Roller of 100 ± 1 mm diameter, 10 ± 0.5 kg mass, and approximately 200 mm length; and
- d) Balance, accurate to 1 mg.

13.2.2.3 Reagents — Distilled or de-ionized water at a temperature of $27 \pm 3^{\circ}\text{C}$

13.2.2.4 Preparation of test pieces — Cut ten square test pieces of each side 12.5 cm from each specimen, free from folds, wrinkles or other blemishes.

13.2.2.5 See that the lower edge of the cylinder and the surface of the rubber backing sheet are dry before a fresh test piece is clamped in position.

13.2.2.6 Duration of test — Test time is the time that elapses between the moment at which the liquid enters into contact with the test piece and the beginning of drying. It is normally 60 seconds; other recommended times are given in Table 3.

TABLE 3 RECOMMENDED TIMES

TIME	SYMBOL OF TEST TIME	INTERRUPTION OF THE CONTACT BETWEEN LIQUID AND TEST PIECE AFTER	DRY AFTER
s		s	s
300	C 300	285	300
120	C 120	105	120
60	C 60	45	60
30	C 30	20	30

- a) A test time of 60 seconds is suitable for most medium and well-sized papers and is known as *one-minute Cobb test*. For very hard-sized papers and boards, it may be advisable to increase the time of exposure to water to 4 minutes 45 seconds and to blot off the water at the end of a further 15 seconds. This is known as the *five-minute Cobb test*. The *one-minute Cobb test* is taken as the standard form of test in all cases except where otherwise stated.

**AMENDMENT NO. 1 AUGUST 2004
TO
IS 1060 (PART 3) : 1969 METHODS OF
SAMPLING AND TEST FOR PAPER AND ALLIED
PRODUCTS, PART 3**

(Page 6, clause 7.3.1, lines 3 and 6) — Substitute ‘15°’ for ‘5°’.

(CHD 15)

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AMENDMENT NO. 3 AUGUST 1978
TO
IS : 1060 (Part I)-1966 METHODS OF SAMPLING
AND TEST FOR PAPER AND ALLIED
PRODUCTS : PART I
(Revised)

Alterations

(Page 11, clause 6.0) — Substitute the following for the existing clause:

‘6.0 General — Select 10 sheets at random and cut from each sheet a test piece of size 25 × 25 cm. Weigh the sheets individually, and calculate the substance of each test piece. Report both the individual values and the mean.’

(Pages 11 and 12, clauses 6.2 to 6.4) — Substitute the following for the existing clauses:

‘6.2 Procedure — Carry out the determination on 10 sheets selected at random. Take a test piece of size 25 × 25 cm from each sheet (see Note). Measure the sides correct to 0.5 percent for each dimension. Determine the area of the test piece, correct to the nearest 0.25 percent of the area. Weigh the test piece correct to 3 significant figures. Conduct the determination on all the test pieces.

NOTE — The use of a template of equivalent accuracy is permitted.

6.3 Calculation

$$\text{Substance in g/m}^2 = \frac{10\,000\ m}{a\ b}$$

where

m = mass in g of the test piece,

a = length in cm of the test piece, and

b = width in cm of the test piece.

6.4 Report — Report the individual test results and the mean.

AMENDMENT NO. 4 OCTOBER 1979
TO
IS : 1060 (Part I)-1966 METHODS OF SAMPLING
AND TEST FOR PAPER AND ALLIED
PRODUCTS: PART I
(Revised)

Alteration

(Pages 26 to 30, clauses 13.2.2 to 13.2.3.8) — Substitute the following for the existing clauses:

‘13.2.2 Test for Water Penetration (Cobb Test)

13.2.2.0 General — The resistance to water penetration is determined by the quantity of water absorbed by a paper or a board when one of its faces is placed in contact with this liquid, under the conditions laid down in this method, and to express the result in gram per square metre stating the duration of the test.

13.2.2.1 Apparatus — Any type of apparatus, which permits the following may be used:

- a) An immediate and uniform contact of the water with the part of the test piece submitted to the test, and
- b) A rapid withdrawal of the test piece without the risk of contact with the water outside the test area by means of a suitable fixing device which makes it possible to maintain the times envisaged in Table 3.

All the components constituting the apparatus which are in contact with the water or may have contact with it shall be made from materials not liable to be affected by the liquid.

The apparatus for this test consists of a short, metal cylinder with a cross-section of 100 cm² (11.29 cm internal diameter) and height about 5 cm, capable of being clamped on to the surface of the test piece. It is necessary initially to check the internal diameter of the cylinder. A cylinder of a different diameter may be used provided a suitable correction is made for the difference in area. The thickness of the wall of the cylinder is not important, but may conveniently be about 6 mm; the lower edge shall be machined smooth. The paper to be tested is placed on a base-board, backed by a piece of sheet rubber. The cylinder is then placed on top of the paper and clamped down firmly.

- b) The Cobb test as described above is not suitable for papers which are completely penetrated by water in less than one minute. In such cases, the total time of test may be reduced to 30 seconds. If the test piece is penetrated even within 30 seconds, a wad of test pieces is treated and weighed as one. Use of several sheets is not desirable and should be adopted only when absolutely necessary, for it is by no means certain what relationship the water absorption of a wad of sheets bears to that of a single sheet. The number of test pieces used and the time of the test shall be reported in such a case.

13.2.2.7 Procedure

- a) *Wetting* — Weigh the test piece correctly to 1 mg and place it on the rubber backing sheet with the surface to be tested uppermost. Place the cylinder on the sample and clamp sufficiently firmly to prevent any leakage of water between it and the test piece. Pour water into the cylinder to a depth of 1 cm and start the stop-watch immediately. If, for example, a test time of 60 seconds has been selected, then after 45 seconds, pour off the water, taking care to see that no water gets on to the remaining surface of the test piece.
- b) *Blotting* — Remove the test piece and place it, with the test side uppermost, on a sheet of blotting paper (13.2.2.2) previously positioned on a flat, rigid surface. Exactly 60 seconds after the commencement of the test, place a second sheet of blotting paper on top of the test piece and remove the excess water, using the hand roller (13.2.2.2) with two rollings (once forward and once back) without exerting any pressure on the roller.
- c) *Weighing* — After blotting, weigh the specimen immediately and quickly, correct to 1 mg, so that the increase in mass due to penetration of water may be determined before loss by evaporation occurs.

13.2.2.8 Rejection of test pieces — Test pieces which show excess water after blotting (which is indicated by the gloss of the surface), or which have been penetrated by water shall be rejected. If the number of rejects exceeds 20 percent, reduce the time of test until a satisfactory result is obtained, the minimum test time being 30 seconds.

13.2.2.9 Number of readings — The top-side and the wire-side are tested from separate test pieces since the two sides may differ in their penetration. Make five determinations for each side.

13.2.2.10 Expression of results — Calculate for each face:

- a) the mean of the results obtained and express it in gram per square metre to the first decimal, and

b) the standard deviation.

Indicate the number of determinations. If the faces are not identifiable give the mean and the standard deviation of the grouped results. Report as cobb value the mean of 10 determinations (see 13.2.2.9), using a standard notation, for example, Cobb₁₀ (value in gram per square metre).

13.2.2.11 Test report — The test report should give the results obtained; it should, among other things, mention optional or any other details of operation not provided for in the standard, incidents which are susceptible of having affected the results and the number of rejected test pieces and reason for rejection.

NOTE — If the method is used with other liquids, they should be stated in the test report. See that the vapour pressure of the liquid being considered does not falsify the results, and that the material is resistant to any possible corrosion that might be provoked by the liquid.

(CDC 15)

**AMENDMENT NO. 5 NOVEMBER 2011
TO
IS 1060 (PART 1) : 1966 METHODS OF SAMPLING AND
TEST FOR PAPER AND ALLIED PRODUCTS : PART 1**

(Revised)

[Page 11, clause 6.0, line 2 (see also Amendment No. 3)] — Substitute ‘minimum 20×25 cm’ for ‘ 25×25 cm’.

(Page 13, clause 9.0, line 5) — Substitute ‘ $105 \pm 2^{\circ}\text{C}$ ’ for ‘ $103 \pm 2^{\circ}\text{C}$ ’.

(Page 15, clause 9.3.2, line 6) — Substitute ‘ $105 \pm 2^{\circ}\text{C}$ ’ for ‘ $103 \pm 2^{\circ}\text{C}$ ’.

(Page 15, clause 9.3.3, line 3) — Substitute ‘ $105 \pm 2^{\circ}\text{C}$ ’ for ‘ $103 \pm 2^{\circ}\text{C}$ ’.

(Page 20, clause 12.3.3) — Substitute the following for the existing:

‘*Test Specimens* — They shall be strips cut accurately and parallel to within 0.1 mm, with clean edges, in each principal direction of the paper, and over 180 mm, preferably 200 mm long. The width shall be 15 to 25 mm. The specimens shall be conditioned. They shall be free from abnormalities, water-marks, creases and wrinkles.’

(Page 22, clause 12.3.5.2) — Add the following at the end of this clause:

‘Tensile index = Breaking length \times 0.009 8’.

(Page 23, clause 12.5.4) — Add the following at the end of this clause:

‘Burst index = Burst factor \times 0.098’.

(Page 23, clause 12.6.2, line 2) — Substitute ‘15 mm \pm 0.1 \times 100 mm’ for ‘15 mm wide and 97 mm long’.

(Page 24, clause 12.7.2, first line) — Add ‘of size 43 ± 0.2 mm \times 63 ± 0.15 mm’ in between the words ‘piece’ and ‘with’.

(Page 25, clause 12.7.5) — Add the following at the end of this clause:

‘Tear index = Tear factor \times 0.098’.

(CHD 15)

Indian Standard
**METHODS OF SAMPLING AND
TEST FOR PAPER AND
ALLIED PRODUCTS: PART I**
(Revised)

0. FOREWORD

0.1 This Indian Standard (Part I) (Revised) was adopted by the Indian Standards Institution on 21 May 1966, after the draft finalized by the Paper Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was first published in 1956. Suggestions were received to modify sampling procedure prescribed in the specification. In this revised version, sampling procedure, method of test for moisture determination and the method for finding rosin sizing have been modified. Method of test for oil absorbency and alternate method for water penetration have been included. Also, some other minor changes have been made in other methods of test to align them with the new developments in the industry.

0.3 The production of paper has increased considerably in the last few years, but the per capita consumption of paper in our country is still one of the lowest as compared to other countries. The production of paper is expected to be roughly doubled in the next five years. It is expected that this standard will be of assistance to the manufacturers for controlling the quality of their products and to the consumers for testing that they get a material of acceptable quality.

0.4 In the formulation of this standard, due weightage has been given to international co-ordination among the standards and practices prevailing in different countries and equipments available in this field in the country.

0.5 This standard gives some of the general methods of test, used more commonly, for paper and allied products. Some other methods of test for special purposes have been covered by IS: 1060 (Part II) - 1960*.

0.6 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS: 2-1960†.

*Methods of sampling and test for paper and allied products, Part II.

†Rules for rounding off numerical values (revised).

1. SCOPE

1.1 This standard prescribes the methods of sampling and test, which are common to several detailed Indian Standard specifications for paper and allied products. It covers method of sampling, preliminary examination of consignment, conditioning and the methods of test for the determination of the following:

- a) Substance or ream weight,
- b) Thickness,
- c) Bulk,
- d) Moisture content,
- e) pH value,
- f) Ash,
- g) Tensile strength and stretch,
- h) Breaking length,
- j) Bursting strength,
- k) Folding endurance,
- m) Tearing resistance,
- n) Sizing,
- p) Resistance of writing papers to feathering,
- q) Water penetration,
- r) Water absorbency,
- s) Gloss,
- t) Opacity,
- u) Oil absorbency, and
- v) Fibre composition (furnish).

1.2 Should there be any inconsistency between the requirements of this standard and those of the standard for an individual material, the latter shall prevail.

2. TERMINOLOGY

2.0 For the purpose of this standard, the following definitions shall apply.

2.1 Standard Atmospheric Conditions — A relative humidity of 65 ± 2 percent and a temperature of $27 \pm 2^\circ\text{C}$, provided that in a given series of experiments the temperature does not vary by more than $\pm 1^\circ\text{C}$ (*see also* IS: 196-1950*).

*Atmospheric conditions for testing. (Since revised).

2.2 Substance—Weight of a given specimen of paper in grams per square metre under standard atmospheric conditions (*see* 2.1).

2.3 Ream Weight—Weight in kilograms of a ream of paper containing 500 sheets of specified size.

2.4 Bulk—Ratio of the volume of paper measured at standard atmospheric conditions to the volume of an equal weight of water at 4°C and is calculated from the formula:

$$\text{Bulk} = \frac{\text{thickness (microns)}}{\text{substance (g/m}^2\text{)}}$$

2.5 Wire-Side—Side of the paper which was in contact with the wire of the paper machine during the course of manufacture.

2.6 Top-Side—Side of the paper opposite to the wire-side.

2.7 Machine Direction—The direction of paper or board corresponding to the direction of the flow of the stuff on the paper machine.

2.8 Cross Direction—Direction at right angles to the machine direction.

2.9 Tensile Strength—The limiting resistance of a test piece of paper or board submitted to a breaking force applied to each of its ends under the conditions defined in the standard method of test. The tensile strength is generally expressed as breaking length.

2.10 Breaking Length—The calculated limiting length of a strip of paper or board of uniform width, beyond which, if such a strip were suspended by one end, it would break by its own weight.

2.11 Stretch—The extension resulting from the application of a tensile stress up to the point of rupture.

2.12 Bursting Strength—The hydrostatic pressure applied at right angles to the surface at which rupture of a circular area of the paper occurs under prescribed conditions of test.

2.13 Folding Endurance—The number of double folds, in opposite directions, at the same place, which paper, under specified tension, can stand up to the point when it ruptures.

2.14 Tearing Resistance—The average force required to tear a specimen of paper after an initial tear.

2.15 Feathering—The irregular spread of ink on either side of the line drawn with writing ink.

2.16 Moisture Content—Percentage of water contained in the materials as determined by heating to constant weight at $103 \pm 2^\circ\text{C}$.

2.16.1 If the material contains matter, other than water, volatile at $103 \pm 2^\circ\text{C}$, the portion thus volatilizing will get included in moisture content and special tests may be required for determining the moisture and volatile components separately.

2.17 Quality of Reagents — Unless specified otherwise, pure chemicals shall be employed in tests and distilled water shall be used where the use of water as a reagent is intended.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

3. SAMPLING

3.1 Definitions — The following definitions as illustrated in Fig. 1 shall apply.

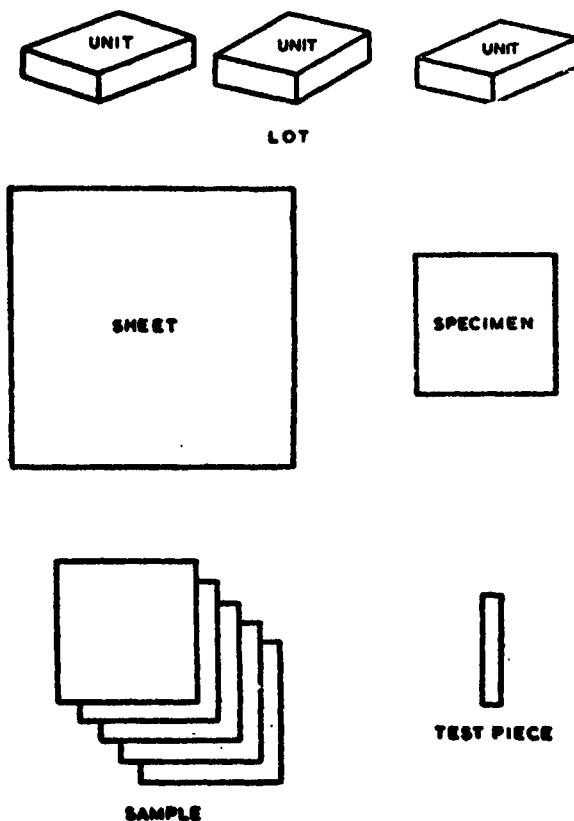


FIG. 1 ILLUSTRATION OF TERMS USED IN SAMPLING

3.1.1 Unit—Quantity of paper of identical specification packed together for convenience in sale, handling and accounting.

3.1.2 Lot—Quantity of paper of identical specification and belonging to the same batch of manufacture.

3.1.3 Sheet—Paper drawn from a selected unit for tests.

3.1.4 Specimen—Sheet cut to given dimensions.

3.1.5 Sample—All the specimens from the same unit or lot.

3.1.6 Test Piece—Piece of paper cut from a specimen for carrying out tests.

3.2 Scale of Sampling

3.2.1 For ascertaining the conformity of the material to the requirements of the relevant material specifications, each lot as defined in 3.1.2 shall be examined separately.

3.2.2 The number of units to be selected from a lot shall be in accordance with col 2 of Table 1 or 2 depending upon the size of the lot as in col 1 of the same tables. Table 1 is for papers of substances up to 250 g/m² and Table 2 is for papers of substances above 250 g/m².

TABLE 1 SCALE OF SAMPLING FOR PAPERS OF SUBSTANCES UP TO 250 g/m²

NUMBER OF UNITS IN THE LOT	NUMBER OF UNITS TO BE SELECTED
(1)	(2)
Up to 5	All
6 to 100	5
101 „ 300	8
301 „ 500	13
501 and over	20

TABLE 2 SCALE OF SAMPLING FOR PAPERS OF SUBSTANCES ABOVE 250 g/m²

NUMBER OF UNITS IN THE LOT	NUMBER OF UNITS TO BE SELECTED
(1)	(2)
Up to 5	All
6 to 300	5
301 „ 500	8
501 „ 1 000	13
1 001 and over	20

3.3 Selection of Units

3.3.1 If the lot is composed of packages (bales or bundles) each containing a number of units in it, at least 20 percent of the packages, subject to a minimum of two, shall be selected at random. From the selected packages, approximately the same number of units shall be selected at random so as to give the requisite number of units required in 3.2.2. At each stage selection shall be at random (*see* 3.3.3).

3.3.2 If, however, the lot is directly composed of units, the required number of units shall be selected at random in the first stage according to 3.3.3.

3.3.3 For the purpose of random selection, a random number table shall be used. In case a random number table is not available, the following procedure shall be adopted:

Starting from any unit in the lot, count them in any order as 1, 2, 3,..... up to r and so on, where r is the integral part of N/n (N being the number of units in the lot and n the number of units to be selected). Every r th unit thus counted shall be withdrawn from the lot.

3.4 Selection of Sheets

3.4.1 The total number of sheets to be taken from each unit shall be determined by the number of tests required to be carried out on the lot as a whole or on each individual unit or both as specified in the relevant material specifications.

3.4.2 The same number of sheets shall be taken from each selected unit.

3.4.3 The selection of sheets from a unit shall be done at random, the intention being to ensure that the selected sheets fully represent the unit concerned.

3.4.3.1 When the unit is composed of sheets (for example, a ream), remove the outermost sheets and then select the requisite number of sheets at random.

3.4.3.2 When the unit is not directly composed of sheets (for example, a roll or reel), remove all damaged layers from the outside and three undamaged layers in case of paper and one in case of board. Cut the unit across its full width to a sufficient depth to yield the requisite number of sheets after rejecting the sheets from the outer layers. The size of the sheets, if possible, should be approximately 45 cm square with sides parallel to those of the unit.

3.5 Selection and Cutting of Specimens

3.5.1 The dimensions of the specimens should normally be approximately 90 × 45 cm (in order to allow for their later reduction in the laboratory),

he greater of these dimensions being in the machine direction if this is known. If machine direction is not known, select a specimen approximately 45 cm square.

3.5.2 *When the Sheets Selected have Dimensions Greater than 30×45 cm*

- a) *Paper or board in sheets*—Cut specimens, one from each sheet, varying the position of selection every time.
- b) *Paper or board in reels*—Cut specimens from each sheet corresponding (approximately) to each 40 cm across the width of the reel.

3.5.3 *When the Sheets Selected have Dimensions Less than 30×45 cm But the Surface Area is Not Less than 1000 cm^2* —If the surface of the sheet is greater than 1000 cm^2 , from each sheet, select a specimen in such a manner that its surface area is as near 1000 cm^2 as possible, preferably a little greater. Mark the machine direction if it is not the longer dimension.

3.5.4 *When the Sheets Selected have Dimensions Less than 30×45 cm and the Surface Area is Less than 1000 cm^2* —The sheets themselves constitute the specimen. Select sufficient sheets to provide the required surface area for the determinations.

3.6 Precautions—The following precautions shall be taken in drawing and handling of samples and test pieces.

3.6.1 Care shall be taken to select samples or portions of rolls or reels that are not damaged. It is good practice to discard a few outermost sheets of reams or the first few layers of roll or reel to be sure of obtaining representative samples.

3.6.2 Where portions have to be taken by cutting, they shall be cut across the full width of the undamaged layers.

3.6.3 Samples shall not be taken from an exposed place.

3.6.4 Samples shall be kept flat, free from wrinkles and folds. They shall be protected from exposure to heat, direct sunlight, liquids, varying humidity conditions as well as any other harmful influences.

3.6.5 Samples shall be handled as little as possible and contact with sweated hands shall be strictly avoided. Contact with hands may quite appreciably affect the chemical, physical, optical, surface or other characteristics.

3.6.6 Samples to be tested for moisture shall be placed immediately after sampling in an air-tight container.

3.6.7 Test of strength characteristics shall not be carried out with portions bearing water-marks, creases or any visible imperfections.

3.7 Identification of Specimen

3.7.1 Each specimen shall be provided with identification marks, this being necessary to ensure its recognition beyond all doubts. These marks should be indelible and may be limited to the number of the sampling report and the signature of the sampler. They should be in one corner and as small as possible.

3.8 Number of Tests and Criteria for Conformity—These shall be given in the individual material specifications.

4. PRELIMINARY EXAMINATION OF CONSIGNMENT

4.1 Condition—Check the consignment visually on its receipt to see if it is in sound condition and is free from damage due to improper packing, handling in transit, etc.

4.1.1 Report the condition of the consignment, stating the nature and extent of damage, if any.

4.2 Weight Checking of Lot

4.2.1 Procedure—Weigh the units selected as specified in 3.2.2 on a suitable scale in stacks of convenient size, depending on the capacity and size of the scale. Calculate the weight of the lot on the basis of the weight of the units.

4.3 Visual Examination

4.3.1 Open the reams and the reels in the units selected as under 3.4.2 and test them for the following:

- a) *Reeling*—Examine the reeling to see (1) if it is in one plane and (2) the cores are securely fitted on to reel centres.
- b) *Count*—Verify the number of sheets in the reams to see if they contain the required number of sheets.
- c) *Blemishes*—Examine the material for blemishes, such as specks, pin-holes, patches, creases, folds, cuts and torn sheets and report the defects.
- d) *Size*—Measure, correct to 2.0 mm, the size (length and breadth in the case of ream paper and width in the case of reels) with the help of an accurate metre rule, suitably subdivided. Length in the case of reels is usually calculated from the turnover in sheets from the reel. The width of reels shall not vary more than ± 0.5 percent with a maximum permissible variation of 6 mm. The variation in the measurement of sheets shall be within ± 0.5 percent provided always that, where 0.5 percent is greater than 6 mm, the permissible variation shall be 6 mm and that, where 0.5 percent is less than 3 mm, the permissible variation shall be 3 mm.

5. CONDITIONING

5.1 Since the exact relationship between the moisture content of paper and the results of various tests are unknown, the paper under test shall be conditioned to standard atmospheric conditions (*see* 2.1) in a suitable room or chamber, unless otherwise specified in the method of test.

5.2 Procedure—Suspend each specimen, until equilibrium is reached, in a suitable room or chamber maintained at standard atmospheric conditions (*see* 2.1) so that conditioning atmosphere has free access to all its surfaces, air being so circulated that the humidity and temperature of the room or chamber are maintained uniformly. The specimen shall be deemed to have reached the equilibrium when the results of two weighings at an interval of not less than one hour between weighings do not differ by more than 0.25 percent of the total weight. Most common varieties of paper require 4 hours to reach equilibrium, but some hard-sized papers and water-resistant and other special papers may require 24 hours or longer.

5.2.1 After the specimens for test purposes are conditioned, they shall be handled as little as possible and not breathed on.

6. SUBSTANCE OR REAM WEIGHT

6.0 General—A specimen or a test piece cut to a suitable size of not less than 25 × 25 cm is weighed and the substance or ream weight is calculated. Weight of a paper is expressed either as substance or ream weight (*see* 2. and 2.3).

6.1 Apparatus—A balance sensitive to 0.25 percent of the load applied and so graduated that readings of this degree of accuracy may be made.

6.1.1 A suitably calibrated sheet-weighing balance, designed to indicate the weight of the ream directly when only one sheet of the paper of the given size is weighed on it, may be used.

6.2 Procedure—Use a specimen or cut out a test piece in the form of rectangle to any convenient size of not less than 25 × 25 cm. Measure the sides correct to 0.5 percent for each dimension. Determine the area of the specimen/test piece correct to the nearest 0.25 percent of the area. Weigh the specimen/test piece correct to 3 significant figures.

6.2.1 The use of a template of this accuracy is permitted.

6.3 Calculation

$$a) \text{ Substance in g/m}^2 = \frac{10\,000\,w}{a\,b}$$

where

w = weight in g of the specimen/test piece weighed,
 a = length in cm of the specimen/test piece weighed, and
 b = width in cm of the specimen/test piece weighed.

b) Ream weight in kg for 500 sheets = $\frac{w \ c \ d}{2 \ a \ b}$

where

w = weight in g of the specimen/test piece weighed,
 c = length in cm of the sheet in the ream,
 d = width in cm of the sheet in the ream,
 a = length in cm of the specimen/test piece weight, and
 b = width in cm of the specimen/test piece weighed.

6.4 Report — Weigh one test piece from each sample sheet separately and report the average, maximum and minimum values of the results.

7. THICKNESS

7.1 Apparatus

7.1.1 Micrometer — Properly calibrated dead weight micrometer, fitted with a dial gauge reading correct to 0.01 mm or, alternatively, a micrometer fitted with a dial gauge reading correct to 0.0005 in. A screw micrometer shall not be used on a yielding material like paper.

7.1.2 Sheet-Holder — The device consists of two parallel plane faces, which are parallel to within 0.005 mm and constrained to move apart in the direction perpendicular to their planes. The smaller of the faces, which generally moves, is circular, with 14 to 16.5 mm diameter, corresponding to an area of 160 to 210 mm². The moving member is geared to the micrometer dial gauge indicator reading correct up to 0.01 mm.

7.2 Procedure

7.2.1 For Sheets Having a Thickness of Below 0.25 mm — Take a pack of not less than 5 specimens or test pieces. Cut to a size of 20 × 25 cm. The number of specimens or test pieces in each pack shall be so chosen that all the sheets (*see* 3.4) are represented in the test. Each specimen or test piece to be tested shall be independent of the remainder, that is, one specimen or test piece folded and inserted in the pack to form two or more specimens or test pieces shall not be used. Raise the moving member of the apparatus by means of the lever, introduce this pack and relax the lever gently to enable the moving member to fall down and touch the pack and exert a steady pressure of 1.00 ± 0.10 kg/cm². Test at 5 places, near the edges as well as in the central portion of the specimen or test piece, to check for uniformity of thickness.

7.2.1.1 Report—Take the average of 5 readings as the thickness of the pack. Take mean of the averages of all the packs. Divide the mean of the averages and the highest and lowest values by the number of specimens or test pieces contained in each pack and report these as the average thickness of each specimen or test piece and the range of variation.

7.2.2 For Sheets Having a Thickness of More than 0.25 mm—Test each specimen or test piece singly and test a piece each from all the specimens. Raise the moving member of the apparatus by means of the lever, introduce the specimen or test piece and relax the lever gently to enable the moving member to fall down and touch the specimen or test piece and exert a steady pressure of $1.00 \pm 0.10 \text{ kg/cm}^2$. Test at 5 places, near the edges as well as in the central portion of the specimen or test piece, to check for uniformity of thickness.

7.2.2.1 Report—Take the average of 5 readings on each specimen or test piece as its thickness. Take the mean of the average of all specimens or test pieces. Report the mean of the average, maximum and minimum values of the readings as the average thickness and range.

7.3 Precautions

7.3.1 Before starting, it is essential to see that there is no clearance between the two parallel faces and that when the moving face touches the other parallel plane face, the pointer on the dial is at zero reading. The pointer should be brought to zero position, if necessary.

7.3.2 Care shall be taken to see that the sheets are free from folds and creases.

8. BULK

8.1 Calculation of Bulk—Calculate the bulk as follows:

$$\text{Bulk} = \frac{\text{Average thickness of a single sheet in microns}}{\text{Substance in grams per square metre}}$$

9. MOISTURE CONTENT

9.0 General—The conditioned specimen is weighed and heated to a constant weight to expel moisture. The difference between two weighings gives the moisture content. This method applied to all paper, paperboard and paper products except those containing significant quantities of materials other than water that are volatile at $103 \pm 2^\circ\text{C}$. When it is required to find the moisture content of paper as received, the samples shall not be conditioned.

9.1 Apparatus

9.1.1 Weighing Container—Either a wide-mouth, glass-stoppered weighing bottle, approximately 65 mm in height and 45 mm in diameter, or, for

IS : 1060 (Part I) - 1966

larger specimens, a metal or an air-tight container, preferably provided with a removable wire mesh basket, and of such a size as to accommodate the specimens without their being tightly packed.

9.1.2 Thermometer — Accurate to within 1 deg and having a range of 0 to 150°C (see IS : 2480-1964*).

9.1.3 Drying Oven — Constant-temperature, equipped with means of ensuring adequate temperature control and free access of air.

NOTE — There is danger of local overheating if the samples are exposed to direct rays of unshielded heating elements.

9.1.4 Chemical Balance — Sensitive to 1 mg for weighing specimens of 2 g and less and to 0.05 percent of the original weight of the specimen for larger specimens.

9.1.5 Desiccator — Anhydrous alumina (indicating grade) is the most suitable desiccant. Calcium chloride and calcium sulphate are unsatisfactory.

9.2 Test Specimen

9.2.1 When the amount of moisture is determined for the purpose of calculating the results of a chemical analysis of paper on a moisture-free basis, use test specimens weighing not less than 1 g and preferably not more than 2 g each. Take care that when weighed, the specimens for moisture determination are in moisture equilibrium with the specimens for chemical analysis. When moisture is determined for the purpose of calculating the amount of moisture in a shipment of paper, obtain test specimens weighing not less than 50 g each.

9.2.2 When the amount of moisture is determined for paperboard or containers which are to be tested for other physical properties, take test specimens weighing approximately 50 g, representative of the material being tested. Cut specimens from unsealed sections of containers, and if possible, from unprinted ones.

9.3 Procedure

9.3.1 In sampling a shipment for moisture determination, take extreme care to avoid any change in moisture content during sampling. Handle large specimens with clean, dry, rubber gloves. Transfer the specimens to the weighed container as soon as they are withdrawn and close the container immediately. If a delay of a few seconds in transferring the specimen to the container is unavoidable, keep the specimen covered on both sides with several adjacent layers of the paper from which it was withdrawn

*Specification for general purpose glass thermometers.

until ready to place it in the container. Unless the specimen is to be spread out in the oven, avoid filling the container tightly. Weigh the specimen in the closed container to obtain its net weight.

9.3.2 For large specimens, unless the container has a removable basket, remove the specimens from the container in which they were weighed, spread them in a tray, preferably made of wire mesh which will permit free circulation of air around them, and place them together with the original containers in the oven. Heat for about 2 hours at $103 \pm 2^\circ\text{C}$. Replace the specimens in the original container and close it without removing the specimens from the oven, if possible. Let the closed container and contents cool at room temperature and weigh.

9.3.3 Place small specimens in the drying oven without removing them from the weighing bottle. Remove the stopper of the bottle, heat for about 1 hour at $103 \pm 2^\circ\text{C}$, close the bottle in the oven, cool to room temperature in a desiccator, loosen the stopper momentarily to adjust any change in air pressure and weigh.

9.3.4 Repeat the periodic drying and weighing of the specimen until the difference in weight between two successive weighings is not more than 0.1 percent of the weight of the specimen.

9.3.5 Make all weighings with the cover on the container and weigh to within 0.05 percent of the total weight of the original specimen.

9.4 Calculation—Calculate the moisture content as percentage on original weight of the material as follows:

$$\text{Moisture content, percent by weight} = 100 \frac{W - w}{W}$$

where

W = original weight of the conditioned specimen before drying,
and

w = weight of the specimen after drying.

9.5 Report—Report the moisture as the percentage loss in original weight of the specimen to the nearest 0.1.

9.6 Precision—The percentage results of duplicate determinations of moisture made at the same time should agree within 0.2.

10. pH VALUE

10.0 General—The following method is suitable for the regular run of commercial and industrial papers, the water extracts of which are normally acidic and usually buffered. It is not adequate for determining the pH of unbuffered and neutral papers, such as insulating papers, which require a

IS:1060 (Part 1) - 1966

more accurate method for eliminating error due to the absorption of carbon dioxide by the water extract during its preparation and testing.

10.1 Apparatus — The following apparatus is required.

10.1.1 Electrometric pH Meter — Any standard pH meter. Calibrate against standard buffer solutions at two pH values (see 10.2.1).

10.1.2 Glassware — As required under 10.3, neutral and resistant to acids and alkalis.

10.2 Reagents — The following reagents are required.

10.2.1 Buffer Solutions — Two standard solutions, one with pH 4 and the other with pH 9.

10.2.2 Distilled Water — pH 6.0 to 7.2.

10.3 Procedure — Cut or shred about 1 g of the specimen in a 125 ml conical flask fitted with a ground-glass air condenser and add up to 20 ml of boiling distilled water in small portions till the paper is wetted. Add another lot of 50 ml of distilled water, fit the reflux air condenser and digest, with occasional shaking, at 98 to 100°C for one hour. At the end of the digestion, cool to 45 to 40°C with the air condenser in position and its top covered by a small beaker. Remove the air condenser, shake the flask thoroughly, close it tightly with a clean rubber stopper, set aside in a cold water-bath and cool to room temperature. Determine the pH of the supernatant liquid with the pH meter. Make at least two determinations on test pieces from two separate specimens and, if the value differs by more than 0.4, repeat with two fresh specimens. Reject the highest and the lowest and report the average.

10.3.1 Excessive contact with air shall be avoided.

11. ASH

11.0 General — Though all fibrous materials used for the manufacture of paper and allied products have an inherent ash, it is generally small. The percentage of ash is, therefore, taken to be an index of added mineral matter or loading. A known weight of the specimen is burnt, ash weighed and the percentage calculated.

11.1 Procedure — Tear about 1 g of the specimen into small shreds and place in a previously weighed crucible. Again weigh. Heat carefully over a Bunsen burner to ensure that the paper burns quietly until it is completely charred. Transfer the crucible into a muffle furnace at $800 \pm 25^\circ\text{C}$ and heat until all the carbonaceous matter is burnt off. Cool the crucible in a desiccator, weigh and repeat the operation till the weight is constant.

11.2 Calculation—Calculate the ash as percentage on the original weight of the material as follows:

$$\text{Ash, percent by weight} = 100 \frac{w - X}{W - X}$$

where

w = weight in g of the crucible and the ash,

X = weight in g of the crucible, and

W = weight in g of the crucible and the material.

11.3 Report—Make the determination on three specimens and report the average, maximum and minimum of the results.

12. STRENGTH

12.0 General—Paper is not uniform in structure. Its properties vary from sheet to sheet and also from place to place in the same sheet. To get a true estimate of a particular strength property, a number of tests has to be made. The test pieces should be cut in direction parallel to that of the machine and cross direction of the paper. The determination of strength may relate to (a) tensile strength and stretch (or elongation), (b) breaking length, (c) bursting strength, (d) folding endurance, and (e) tearing resistance.

12.1 Determination of Machine Direction

12.1.0 Any one of the following three methods shall be employed for determining the machine direction of the paper.

12.1.1 Method 1—For the purpose of identification, draw a straight line on the paper under test. Cut out a circular test piece, about 50 mm in diameter, taking care to see that the test piece and the adjacent parts of the paper from which the test piece is cut, carry the line. Float the test piece on water and note the direction of curl. The axis of the curl will be found parallel to the machine direction of the paper.

12.1.2 Method 2—Burst a test piece in a manner similar to that employed while testing for bursting strength (see 12.5). The chief line of rupture is at right angles to the machine direction of the paper.

12.1.3 Method 3—Cut two test pieces, 15 × 150 mm each in directions at right angles to each other. Superimpose and hold the pieces together at one end so that the edge is in the horizontal. The free ends of both the pieces will bend over towards the vertical. Observe the angle between the pieces. Place the bottom piece on top and note the new angle. The cross-direction strip is the one which is at the bottom when the angle is greater.

12.2 Determination of Top-Side and Wire-Side

12.2.0 Any one of the following two methods shall be employed for determining top-side and wire-side of the paper.

12.2.1 Method 1—Examine the paper under oblique illumination, if necessary, using a low power lens. The side which shows a mesh-like structure is the wire side. If the mesh structure cannot be ascertained in this way, moisten the paper with water or dilute caustic soda solution. This causes the fibres to swell and usually makes the mesh structure more clearly visible.

12.2.2 Method 2—Cut several test pieces 2 to 3 cm wide and 5 to 10 cm long so that the cross direction of the paper runs with the length of the test piece. Mark the test pieces on the same side. Clamp two of them breadthwise in the middle in a narrow clamp so that when the test pieces are viewed from either side, only one identification mark is visible. Immerse the test pieces in water as shown in Fig. 2. Observe the direction in which the test pieces curl in the water. The convex side is the wire side.

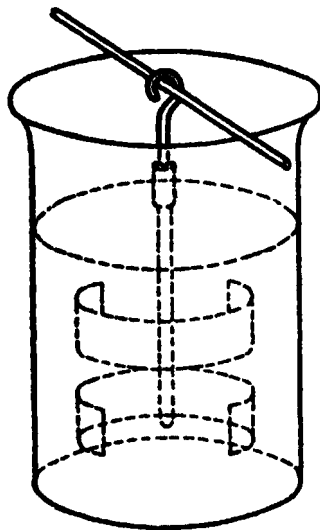


FIG. 2 DIAGRAM SHOWING THE DIRECTION OF CURL OF PAPER IN DETERMINING TOP-SIDE AND WIRE-SIDE

12.3 Tensile Strength and Stretch (or Elongation)

12.3.0 General—This test is performed to determine the resistance to pull of paper and the percentage elongation the paper undergoes before

fracture. Tensile strength is greater in the machine direction than in the cross direction. Elongation is usually less in the machine direction than in the cross direction. Tensile strength and stretch is measured by the tensile pull necessary to break a strip of paper and allied products.

12.3.1 Apparatus — Any apparatus which is capable of acting on the test piece at the defined rate and permitting the tensile force at the moment of failure to be read to an accuracy of 1 percent may be used.

12.3.1.1 Two clamps whose centres shall be in the same plane parallel to the direction of motion of the applied stress and so aligned that they will hold the test specimen in one place throughout the test without slippage. At the start of the test, the edges of the jaws of the clamps shall be 180 ± 10 mm apart, except that for coarse papers, such as building papers, and for paperboards over 0.30 mm thick, the distance may be 150 to 200 mm.

12.3.1.2 Means of applying a predetermined initial tension to the test specimen while it is being clamped.

12.3.1.3 Means of applying a gradually increasing load to the test specimen until it breaks, the increase being such that the additional load applied each second is not different by more than 5 percent from the additional load applied in the previous second. The rate of loading should be so adjusted that failure of the test piece can be obtained in a mean time of 20 ± 5 seconds.

12.3.1.4 Means of indicating the elongation of the test specimen to an accuracy of within 0.5 mm and applied load to an accuracy of ± 2 percent at the instant of complete fracture.

12.3.2 Calibration

12.3.2.1 Level the machine accurately in both the principal directions and clean the mechanism to ensure that it moves freely. Apply various dead weight loads to the clamp actuating the indicating mechanism and note the scale readings when the load and mechanism come gently into an equilibrium position. This may be done conveniently with the pendulum-type of tester by wedging up the pawls holding the pendulum with a small piece of paper, folded double, suspending the test weight to the upper clamp and allowing the pendulum to come to equilibrium from the direction in which it moves when the load is applied to it. The lower jaw should not be used for supporting the test weights during calibration. Make a record of deviations from the indicated readings and apply corresponding corrections to the test results. In general, the calibration of the scale should be checked at three or four widely-spaced points, and, if appreciable errors are found, enough calibration points should be used to allow construction of a correction curve. It is recommended that the instrument shall have been calibrated within one month prior to the test.

12.3.2.2 For calibrating the stretch-indicating mechanism, clamp the pendulum at zero, set the lower clamp near the upper clamp and set the stretch indicator at zero, with the trigger mechanism operating the stretch indicator adjusted to and engaged with the lower clamp. With inside vernier calipers measure the distance between the clamps to the nearest 0.2 mm. Move the lower clamp down a little distance and again measure the distance between the clamps. The indicated reading should correspond to the difference between the two vernier readings. Repeat the same procedure for various points along the elongation scale. An alternative method of calibration is given below:

Grip a heavy rubber strip between the clamps of the tester and compare the changes in the indicated readings with the distances between the clamps, as measured with vernier calipers, using a rule with squared ends to extend the range of the calipers, if required. If necessary, prepare a calibration table or chart for applying any corrections. To ensure that the stretch-indicating mechanism is stable, place the pendulum in position about halfway up the scale, set the stretch indicator at some intermediate position and then jar the instrument slightly and note whether the stretch indicator moves. If it does, either the stretch mechanism shall be counterbalanced or the spring friction holding it in position shall be increased. Otherwise, a serious error may be caused in the reading by the jar. This occurs especially when a strong specimen breaks under test.

If necessary, adjust the overhang on the trigger-release mechanism so that, immediately after the strip breaks, the indicator is no longer actuated. Unless the apparatus is altered in any vital way, there is no need to repeat the calibration once it has been established; but if the stretch-indicating mechanism is not counterbalanced, it should be tested for stability to jarring from time to time.

12.3.3 Test Specimens—They shall be strips cut accurately and parallel to within 0.1 mm, with clean edges, in each principal direction of the paper, and over 200 mm, preferably 250 mm long. The width shall be 10 to 50 mm. The specimens shall be conditioned. They shall be free from abnormalities, water-marks, creases and wrinkles.

12.3.4 Procedure

12.3.4.1 By an initial experiment the rate of application on tensile force is selected which causes failure in a mean time of 20 ± 5 seconds.

For weak papers, such as tissues, paper towels, newsprint, etc, requiring a breaking load of 2.3 kg or less, have the machine speed so adjusted that fracture occurs in not less than 5 nor more than 15 seconds. For other papers and paperboards requiring a breaking load of not more than 13.6 kg, use a constant rate of loading of about 0.45 ± 0.15 kg per second.

For papers and paperboards requiring a breaking load of more than 13.6 kg, adjust the machine speed so that fracture occurs in not less than 30 nor more than 45 seconds.

12.3.4.2 Tightly grip one end of each strip in the upper clamp after placing the strip loosely in the lower clamp and checking its alignment. Then apply an initial stress of 0.23 kg to each strip before clamping except for papers having a tensile strength of less than 2.3 kg. In the latter case, subject two or three test specimens from the same sample of paper to a preliminary tensile test and apply an initial tension equal to 10 ± 2.5 per cent of the average break load so determined to each strip while being clamped. With the apparatus in which the test specimen is clamped vertically, this is conveniently done by temporarily flipping a suitable weight to the lower protruding end of the strip before tightening the lower clamp. Tightly clamp both ends of the strip and apply the load.

Note the readings on the load scale and the stretch indicator at the instant of break. Reject readings from individual strips if the strip, slips or breaks in or at the edge of clamps. Record the result of each individual reading to two significant figures in case of stretch and three significant figures in case of tensile strength. Test at least ten, preferably twenty, strips cut in each principal direction of the paper.

If the mean value of the lowest and the highest reading differs from the average of all the readings by more than 5 percent, test more specimens until there is agreement within this limit. In the case of an irregular sheet of paper, it may be necessary to conduct a much larger number of tests than ten. An isolated very high or low result, which is not repeated in duplicate, shall be discarded when a consistent average is obtained without the abnormal reading.

NOTE — In testing creped paper for stretch, follow the same procedure except, if necessary, reducing the length of the specimen to enable the elongation to be within the range of the indicating mechanism, and apply no more initial tension to the test strip before clamping than is required to straighten it.

12.3.5 Report

12.3.5.1 Results obtained on strips cut in the machine direction shall be reported as tensile breaking strength or stretch, machine direction, and results obtained on strips cut in the cross direction shall be reported as tensile breaking strength or stretch, cross direction. The average value of stretch results on the individual strips shall be reported as a percentage of the length between the clamps, to one decimal place. The average value of the breaking load results shall be reported in kilograms per 15 mm width to the nearest 2 percent of the total reading. The average, maximum and minimum tensile strength for each of the principal directions of the paper shall be reported.

12.3.5.2 A complete report requires, in addition, the following:

- a) Maximum and minimum results and the number of strips tested;
- b) The length of the test specimen, that is, the distance between clamps at the start of the test;
- c) The width of the test specimen; and
- d) A statement of the rate of loading used.

12.4 Breaking Length — The tensile strength is often expressed as the breaking length. This is very useful as an estimate of the ratio of the tensile strength to the substance and is obtained by the following calculation.

12.4.1 Calculation

Breaking length in metres

$$= \frac{\text{Tensile strength in kg per cm width of test piece} \times 100\,000}{\text{Substance in g per square metre}}$$

12.5 Bursting Strength

12.5.0 General — The popularity of bursting strength test depends not only on the ease with which the test is made but also on the combination of strength and the toughness which it measures and which serves as a measure of the serviceability of paper in various applications. It has the disadvantage, however, that it depends in a complicated way on the machine direction, tensile strength, stretch and size of the burst area. Also, it does not give any indication of the cross-direction tensile strength. Bursting strength is measured by the pressure developed behind a circular rubber diaphragm when it is forced through the paper so as to burst it.

12.5.1 Equipment — A tester, in which testing is done by hydraulic pressure communicated through the medium of glycerine or by compressed air to a pure gum rubber diaphragm in contact with the paper, shall be used. The gauge used shall be so chosen that the individual reading shall not be less than 25 percent or more than 75 percent of the total indicated capacity of the gauge.

NOTE — The use of hand operated hydraulic type tester is not recommended as a standard practice.

12.5.1.1 Diaphragm — The diaphragm used in the equipment shall be such that it does not materially affect the bursting pressure and shall be between 0.35 mm and 0.45 mm thick. The rubber sheet used shall be pure gum vulcanizate containing not less than 95 percent by volume of first quality smoked sheet rubber; the only ingredients in the mix, apart from rubber, shall be those necessary to effect correct vulcanization and resistance to premature aging at normal temperatures. The pressure required to bulge the diaphragm 5 mm above the top plane of the lower clamping surface of the test instrument shall be not more than 0.07 kg/cm².

The diaphragm shall be clamped with its outside edge under the lower clamping plate and shall have been renewed less than six weeks prior to test.

12.5.2 Procedure—Clamp the test piece firmly over the diaphragm without slippage during the test between two annular, plane, unpolished (matt) surfaces of 30 mm internal diameter. Run the machine so that the pressure increases at a uniform rate of approximately 0.75 kg/cm² per second until the test piece bursts. Note from the pressure gauge the pressure in kilograms per square centimetre at which the test piece bursts. Take two readings with each sample sheet, one with the wire-side uppermost and the other with the top-side uppermost.

NOTE—A rate of 120 revolutions per minute in the glycerine-operated machine is usually satisfactory.

12.5.3 Report—Report the type of the tester used and give the average, maximum and minimum values of the reading for each side separately.

12.5.4 Burst Factor—Used for comparing two papers with regard to their bursting strength and is calculated as follows:

$$\text{Burst factor} = \frac{\text{Bursting strength in g/cm}^2}{\text{Substance in g/m}^2}$$

12.6 Folding Endurance

12.6.0 General—Folding endurance test is the best available criterion for testing the serviceability of paper that is creased or folded repeatedly. This test gives information about certain properties of paper, such as durability, which cannot be obtained by other tests. A strip of paper is continuously folded till it breaks, the number of double folds giving the folding resistance.

12.6.1 Equipment—‘Schopper’ type, double-fold testing machine is recommended. The machine is driven by motor or countershaft with the help of a friction pulley effecting 90 to 120 double folds per minute. When the machine runs, the slotted folding blade slides back and forth in reciprocating motion between creasing rollers. The clamps are under spring tension which can be varied. The number of times the paper goes through each double fold (back and forth) is counted on a rotating disc known as counter which is designed to count up to 10 000 double folds.

12.6.2 Test Pieces—These are cut from machine and cross directions, exactly 15 mm wide and 97 mm long. At least one test piece in each direction shall be cut from each sample specimen.

12.6.3 Procedure—Slip the test piece into the slotted folding blade and hold it in clamps placed 90 mm apart. Adjust the spring tension in such a manner that it is not less than 770 g when the clamps are farthest apart

IS: 1060 (Part I) - 1966

and not more than 1 050 g when they are nearest to each other. Start the machine and keep the test piece folding back and forth until breakage occurs. Perform the test on all the test pieces.

12.6.4 Precautions— Before putting test pieces on the machine, see that the machine is in locked position. Bring the counter to ZERO position, fasten the paper securely in clamps and make sure that it is free from creases and folds.

12.6.5 Report— Report the average, maximum and minimum of the number of double folds that the test pieces are able to sustain up to rupturing point in each direction separately.

12.6.6 Limitation of the Method— The variability of the results on a single specimen is such that the average of 10 tests may only be relied on to be within 15 percent of the true mean. Two average results cannot be classed as significantly different unless they are 20 percent of their mean apart. If less than 10 tests are taken, these limits shall be extended. Therefore, if necessary, more than one test piece, in each direction, shall be taken from each specimen so that a minimum of ten determinations are made in each direction.

12.7 Tearing Resistance

12.7.0 General— The tearing resistance is usually greater in the cross direction than in the machine direction.

12.7.1 Equipment— Ballistic type of tear-tester, such as the Elmendorf, is recommended. The machine is provided with two clamps, the one fixed and the other carried on a sector-shaped pendulum, suspended from a column by means of a frictionless bearing located near the apex of the sector. On releasing the pendulum, the centre tongue is subjected to the load of pendulum recorded through a spring loaded friction pointer on the circumferential scale marked on the pendulum.

12.7.2 Test Piece— Accurately cut the test piece with a template in such a manner that two parallel slits form a centre tongue giving a double tear. At least one test piece in each direction shall be taken from each specimen.

12.7.3 Procedure— Hold outer tongues of the test piece in a fixed clamp and the centre tongue in the movable clamp. Release the pendulum and note the load necessary to continue the tear. The tests may be made either on a single test piece or in packs of two or more test pieces so adjusted that the reading is not less than 25 percent and not more than 75 percent of the capacity of the instrument. The tearing resistance shall be tested separately for machine and cross direction.

12.7.4 Report— Report the average, maximum and minimum of the readings in each direction separately and state the number of test pieces used for each determination.

12.7.5 Tear Factor — Used for comparing two papers with regard to their tearing strength and is calculated as follows:

$$\text{Tear Factor} = \frac{\text{Tearing resistance}}{\text{Substance}}$$

13. SIZING

13.1 Qualitative Tests — Various sizing materials are used, among which starch, rosin and gelatine are important. The following methods of test are prescribed for identifying these sizing components.

13.1.1 Starch Sizing — Drop on a test piece with a glass rod a weak solution of iodine in potassium iodide, approximately 0.005 N. Alternatively, treat a hot water extract of the paper with the iodine solution. The appearance of a distinct blue colour indicates the presence of starch, the deeper the colour the greater the quantity of starch.

NOTE — A faint colour shall not be taken as evidence of added starch, as in rag pulp it is very difficult to remove starch from the raw materials.

13.1.2 Rosin Sizing — Take a test piece of paper about 200 × 25 mm, pleat it repeatedly, place it in a test-tube and cover it with rectified spirit. Place the test-tube in a water-bath maintained at about 75°C till two-thirds of the rectified spirit has evaporated off. Remove the paper and evaporate the rectified spirit completely. Add 1 ml of acetic anhydride in the tube and dissolve the residue by warming. Cool it and add one drop of sulphuric acid of sp gr 1.53. Formation of a fugitive violet colour indicates the presence of rosin sizing in the paper.

13.1.3 Gelatine Sizing — Cut a small quantity of paper from the specimen and boil for a few minutes in a beaker containing sufficient water to cover the paper. Pour off into a test-tube, cool, add a few drops of 2 percent solution of tannic acid. A flocculent precipitate indicates that the paper has been sized with gelatine. On heating the liquid, the precipitate will coagulate and cling to the sides of the test-tube.

13.1.4 Casein — Make a weak sodium carbonate or sodium borate extract of the paper, filter it off and add dilute acetic acid to the filtrate. Any casein present comes down as a white precipitate; if this is filtered off and washed, it will give a purple coloration on warming with strong hydrochloric acid. If Millon's reagent is used with the neutralized extract, a red colour develops on warming. This reagent is prepared by dissolving 5 g of mercury in 10 ml of concentrated nitric acid and diluting with 50 ml of distilled water after the mercury has been completely dissolved in the acid.

13.2 Sizing Properties

13.2.1 Resistance of Writing Papers to Feathering — For carrying out a quick performance test for resistance to feathering, write on the surface of

IS: 1060 (Part I) - 1966

a test piece with a latem pattern nib and blue-black superior writing ink (conforming to IS:222-1962*). For testing papers which have to stand erasure, such as ledger papers, repeat this test on the same spot after ensuring the original writing. Examine the edges of the writing for definition. The presence or absence of feathering (the irregular spread of ink on either side of the written line) shall be reported. It is indicative of the quality of the sizing.

13.2.2 Test for Water Penetration (Cobb Test)

13.2.2.0 General—A test piece of known area, preferably 100 cm², is brought into contact with water for a specified interval of time and the weight in grams of water absorbed per square centimetre of the surface taken as an index of the water penetration of the paper. The top-side and the wire-side are tested from separate test pieces since the two sides may differ in their penetration.

13.2.2.1 Equipment—The apparatus for this test consists of a short, metal cylinder with a cross-section of 100 cm² (11.29 cm internal diameter) and height about 5 cm, capable of being clamped on to the surface of the test piece. It is necessary initially to check the internal diameter of the cylinder. A cylinder of a different diameter may be used provided a suitable correction is made for the difference in area. The thickness of the wall of the cylinder is not important, but may conveniently be about 6 mm; the lower edge shall be machined smooth. The paper to be tested is placed on a base-board, backed by a piece of sheet rubber. The cylinder is then placed on top of the paper and clamped down firmly.

13.2.2.2 Preparation of test pieces—Cut two test pieces from each specimen, free from folds, wrinkles or other blemishes.

13.2.2.3 The temperature of water is important and shall be maintained at $27 \pm 3^{\circ}\text{C}$.

13.2.2.4 See that the lower edge of the cylinder and the surface of the rubber backing sheet are dry before a fresh test piece is clamped in position.

13.2.2.5 Procedure

- a) **Wetting**—Weigh the test piece correctly to 10 mg and place it on the rubber backing sheet with the surface to be tested uppermost. Place the cylinder on the sample and clamp sufficiently firmly to prevent any leakage of water between it and the test piece. Pour water into the cylinder to a depth of 1 cm and start the stop-watch immediately. After 45 seconds, pour off the water, take care to see that no water gets on to the remaining surface of the test piece. Unclamp and remove the cylinder quickly.

*Specification for ink fluid for general purposes (revised).

- b) *Blotting*—At the end of exactly 60 seconds from the beginning of the test, remove the surplus water from the samples and press tightly with blotting-paper. Excess water remaining after the blotting may easily be seen by the appearance of a highly reflecting area on the surface. If this is the case, further blotting is necessary.

13.2.2.6 *Duration of test*

- a) A test time of 60 seconds is suitable for most medium and well-sized papers and is known as *one-minute Cobb test*. For very hard-sized papers and boards, it may be advisable to increase the time of exposure to water to 4 minutes 45 seconds and to blot off the water at the end of a further 15 seconds. This is known as the *five-minute Cobb test*. The *one-minute Cobb test* is taken as the standard form of test in all cases except where otherwise stated.
- b) The Cobb test as described above is not suitable for papers which are completely penetrated by water in less than one minute. In such cases, the total time of test may be reduced to 30 seconds. If the test piece is penetrated even within 30 seconds, a wad of test pieces is treated and weighed as one. Use of several sheets is not desirable and should be adopted only when absolutely necessary, for it is by no means certain what relationship the water absorption of a wad of sheets bears to that of a single sheet. The number of test pieces used and the time of the test shall be reported in such a case.

13.2.2.7 *Weighing*—After blotting, weigh the specimen immediately and quickly, correct to 10 mg, so that the increase in weight due to penetration of water may be determined before loss by evaporation occurs.

13.2.2.8 *Number of readings*—Make separate determinations on each side of the test piece.

13.2.2.9 *Calculation and reporting of results*

- a) Report the duration of the test and the number of test pieces if more than one piece is used.
- b) The *one-minute Cobb figure* is defined as the amount of water in grams taken up by one square metre of exposed surface in one minute. If the cross-sectional area of the cylinder is exactly 100 cm² and the increase in weight of the paper sample is expressed in centigrams, the *one-minute Cobb figure* would be the same number expressed as grams per square metre.
- c) Report the average, maximum and minimum values for each side separately.

13.2.2.10 Precision—There is no appreciable personal error. When more than three determinations are made, the mean of the determinations is within 10 percent of the true mean. The degree of sizing of two papers may not be taken as significantly different unless difference between the mean values is more than 15 percent.

13.2.3 Alternative Method

13.2.3.0 General—The resistance to water penetration is determined by the quantity of water absorbed by a paper or a board when one of its faces is placed in contact with this liquid, under the conditions laid down in this method, and to express the result in gram per square metre stating the duration of the test.

13.2.3.1 Apparatus—Any type of apparatus, which permits the following may be used:

- a) An immediate and uniform contact of the water with the part of the test piece submitted to the test, and
- b) A rapid withdrawal of the test piece without the risk of contact with the water outside the test area by means of a suitable fixing device which makes it possible to maintain the times envisaged in Table 3.

All the components constituting the apparatus which are in contact with the water or may have contact with it shall be made from materials not liable to be affected by the liquid.

The test surface is a circle of 100 cm²*

13.2.3.2 By way of example, a convenient apparatus would be as follows:

A reservoir, preferably of cylindrical shape, capable of turning about an axis parallel to its opening. This has a test surface of 100 cm²; it includes a raised base and a device which permits the test piece to be fixed by the intermediary of a flexible sheet and a rigid cover supported on the sheet so that the system of fixing ensures a perfect seal.

13.2.3.3 Accessories

- a) Stop watch;
- b) Sheet of blotting paper of 250 g/m² and of 70 mm capillary rise;
- c) Roller of 100 ± 1 mm diameter, 10 ± 0.1 kg weight, approximately 200 mm length and a linear pressure of 0.5 kgf/cm²†; and
- d) Balance, accurate to 1 mg.

*Diameter between 112.5 and 113 mm.

†Alternatively, a press allowing the application of a pressure of 0.5 kg/cm² for two seconds.

13.2.3.4 Reagents—Distilled or *de-ionized water* at a temperature of $27 \pm 3^\circ\text{C}$.

13.2.3.5 Preparation of test pieces—Cut two test pieces from each specimen, free from folds, wrinkles or other blemishes.

13.2.3.6 Procedure—Test time is the time that elapses between the moment at which the liquid enters into contact with the test piece and the beginning of drying. It is normally 60 seconds; other recommended times are given in Table 3.

TABLE 3 RECOMMENDED TIMES

(Clauses 13.2.3.1 and 13.2.3.6)

TIME	SYMBOL OF TEST TIME	INTERRUPTION OF THE CONTACT BETWEEN LIQUID AND TEST PIECE AFTER	DRY AFTER
(s)		(s)	(s)
300	C 300	290	30
60	C 60	50	60
30	C 30	20	30

The order or the nature of the following operations may be varied according to the material used:

- Operate in a conditioned room.
- Weigh the test piece to the nearest milligram (let w_1 be its weight).
- Pour the water into the reservoir of the apparatus to a height of approximately 1 cm.
- Verify that the clamping surfaces are dry.
- Fix the test piece in the apparatus, the surface to be tested being underneath.
- Put the water into contact with the test piece by pivoting the reservoir at 180° round its axis and start the stop watch.
- Approximately ten seconds before the end of the test time required, interrupt the contact of the water with the test piece by turning the apparatus back, raise the lid rapidly and withdraw the test piece.
- At the end of the test time, blot the excess water on the surface tested by placing the sheet between two pieces of blotting paper, the moist side uppermost, and make one pass to and fro with the roller over the whole surface but without pressure in such a manner that it overlaps the edges of the test piece by about 25 mm.

- j) Weigh the test piece to the nearest mg (let w_2 be its weight); the time between the end of the drying and the end of weighing should be less than one minute.
- k) Renew the water for each determination.
- m) Consider only those test pieces which are not penetrated by water. For each of these, the quantity of water retained is the difference between the weights ($w_2 - w_1$) measured in milligrams.

13.2.3.7 Expression of results

Calculate for each face:

- a) the mean of the results obtained and express it in gram per square metre to the first decimal, and
- b) the standard deviation.

Indicate the number of determinations. If the faces are not identifiable give the mean and the standard deviation of the grouped results.

Use the notations:

Cobb	Cobb	Cobb	etc
water 30,	water 60,	water 300,	

13.2.3.8 Test report—The test report should give the results obtained; it should, among other things, mention optional or any other details of operation not provided for in the standard, as well as incidents which are susceptible of having affected the results.

NOTE—If the method is used with other liquids, they should be stated in the test report. See that the vapour pressure of the liquid being considered does not falsify the results, and that the material is resistant to any possible corrosion that might be provoked by the liquid.

14. WATER ABSORBENCY

14.0 General—This test is intended for unsized and absorbent papers, namely, towelling or blotting papers, and consists in allowing water to be sucked up by a strip of the material and determining the rate.

14.1 Procedure—Cut test pieces of 150 × 25 mm size from each direction of each specimen. Make a pencil mark parallel to and 10 mm above one of the shorter edges of each test piece and immerse the strip up to the pencil mark in water at $27 \pm 2^\circ\text{C}$. Note the height in millimetres above the pencil mark to which the water rises in a specified interval of time, the time being taken from the moment of immersion of the test piece in water.

14.2 Report—Report average, maximum and minimum values for each direction separately.

15. GLOSS

15.0 General— This test is performed to find the degree of specular reflectance of papers like supercalendered, imitation art and art.

15.1 Apparatus— The following apparatus is recommended.

15.1.1 Ingersol Glarimeter— for paper of low gloss (15 percent reflectance and below).

15.1.2 Photo-Electric Reflection Meter— for paper of high gloss (15 to 75 percent reflectance) employing an angle of 75°.

15.2 Procedure— Determine the reflectance of each specimen by the method appropriate to the instrument used, utilizing the table supplied with the instrument.

15.3 Report— Report the mean, minimum and maximum results for the top-side and wire-side separately.

16. OPACITY

16.0 General— The method of test described below covers the procedure for measuring the opacity of all kinds of paper and paper products by determining the apparent light reflectance.

16.1 Apparatus

16.1.1 The apparatus shall be capable of measuring the apparent light reflectance as prescribed in this method. It may measure the values separately or give directly the ratio of the apparent reflectances.

16.1.2 Values of apparent light reflectance are relative to the apparent reflectance from magnesium oxide taken as 100 percent. The standard white backing shall have an apparent reflectance of 91.5 percent and the standard black backing shall have an apparent reflectance of not more than 0.5 percent.

16.1.3 Completely diffused illumination from incandescent lamps at a colour temperature of 2400 to 2800° Kelvin shall be used. The direction of viewing shall be not more than 20° from the normal to the surface of the specimen.

16.1.4 Observations shall be made visually or by equivalent means, such as a photo-electric cell with a filter adjusting its sensitivity to that of the human eye.

16.1.5 The instrument shall be calibrated.

16.2 Procedure

16.2.1 Place the test piece first over the standard white backing, then over the standard black backing and then measure the apparent reflectance of the light.

16.2.2 Calculation—The ratio of reflectance over black backing to that over white backing, expressed as a percentage, is the contrast ratio. Calculate the average contrast ratio from determinations on both sides of each test piece.

17. OIL ABSORBENCY

17.0 Outline of the Method—This method consists of measuring the time in which a drop of castor oil produces a uniform translucent spot in paper. It is a measure of the receptivity of paper to printing inks having an oil vehicle, but is suitable only for easily permeable papers, such as news, book and mimeograph.

17.1 Apparatus

17.1.1 Viewing Box—Having an open front, a ground-glass top with a 20 mm hole for observing the specimen, a ground-glass partition parallel to the front side to prevent excess heat from affecting the test results, a 15-watt electric bulb placed at the back of the partition for illuminating the specimen and an adjustable mirror near the bottom of the box and centred on the observation hole in the top of the box.

17.1.2 Separatory Funnel—With a tip approximately 20 mm in length and of such diameter that 25 drops of distilled water delivered at 21°C will have a volume of 1 ml. The funnel is suspended with the end of the tip approximately 45 mm above the test specimen and contains castor oil, the temperature of which is maintained at $27 \pm 2^\circ\text{C}$ during the test.

NOTE—The viscosity of castor oil has been found to vary somewhat. Accordingly, for particular tests, agreement should be reached regarding the specific sample of oil to be used.

17.2 Test Specimens—Select the sample and cut representative specimens, each about 5 cm square.

17.3 Procedure—Place the conditioned specimen over the hole in the top of the box. Let a drop of castor oil (*see* IS:435-1954*) fall from the funnel upon the specimen and start stop watch the instant the drop strikes the specimen. Observe the underside of the specimen and measure the time interval from the instant of contact of the oil with the paper until the spot of oil reaches a uniform and maximum translucency. Covering the spot of oil with a cap having a black interior helps in the determination of the end point.

17.4 Report—Report the maximum, minimum and average time of penetration, to the nearest 5 seconds, for not less than five tests on each side of the paper.

*Specification for castor oil. (Since revised).

18. FIBRE COMPOSITION (FURNISH) QUALITATIVE TESTS**18.1 Colour Reactions for Highly Lignified Fibres, such as Mechanical Wood**

18.1.1 Phloroglucine Solution Test—Make a solution of 1 g of phloroglucine in 50 ml of rectified spirit (conforming to IS:323-1959*) and 25 ml of concentrated hydrochloric acid (conforming to IS:265-1962†). Place a drop of this solution on the test piece. If mechanical wood is present, an intensely red coloration will develop.

18.1.2 Aniline Sulphate Solution Test—A freshly prepared 2 percent (*w/v*) aqueous solution of aniline sulphate containing a drop of sulphuric acid gives a yellow coloration with mechanical wood in the cold.

18.2 Microscopic Examination—This method covers the identification of the kinds of fibres present in paper and paperboard.

18.2.1 Apparatus—The following apparatus is required:

- a) *Microscope*—Compound, equipped with a mechanical stage, Abbe condenser eyepiece and achromatic objective. A magnification of 100 diameters is recommended.
- b) *Dropper*—A glass tube, 6 mm internal diameter, 10 cm long, fitted at one end with a rubber bulb to deliver 0.5 ml.
- c) *Slides and cover glasses*—Slides (25 × 75 mm) and cover glasses (25 × 25 mm) of colourless glass. The slides and cover glasses shall be kept in 50 percent alcohol, or washed with it before use.
- d) *Hot-plate*—With a solid metal top capable of heating to 50 to 60°C.
- e) *Dissecting needles*

18.2.2 Reagent—Herzberg stain.

- a) *Preparation*—Mix 25 ml of an aqueous solution of zinc chloride saturated at 20°C with a solution containing 0.25 g of iodine and 5.25 g of potassium iodide dissolved in 12.5 ml of distilled water. Pour into a narrow cylinder and let stand until clear (12 to 24 hours). Decant the supernatant liquid into an amber-coloured glass-stoppered bottle and add a small piece of iodine to the solution. Avoid undue exposure to light and air. It is necessary to test the stain on a mixture of fibres known to contain about equal proportions of rag, chemical pulp and mechanical wood. If the colour distinction is not satisfactory, either zinc chloride or iodine shall be added till it is satisfactory.

*Specification for rectified spirit (*revised*).

†Specification for hydrochloric acid (*revised*).

- b) *Application* — For staining, apply 3 or 4 drops of the stain to the dried fibres, cover the whole with a cover glass in such a manner as to avoid air bubbles. let stand for 1 or 2 minutes and drain off the surplus with filter paper.

18.2.3 Disintegration of Test Pieces — Shred the test pieces and place in a small beaker. Obtain the approximate weight of the shreds in order to calculate the proper dilution of the disintegrated test pieces. Cover with 1 percent sodium hydroxide solution (*w/v*), bring to boil on a hot plate, decant the liquid and wash twice with distilled water. Cover with 0.5 N hydrochloric acid, let stand for several minutes, decant the acid and wash several times with distilled water. Drain off the water, roll the pieces of paper into pellets gently between the thumb and the fingers, put into a 500-ml Erlenmeyer flask. add a little water and shake vigorously until the water is absorbed by the paper. Add more water and shake, and continue this treatment until the paper is thoroughly disintegrated. Dilute the suspension of fibres by pouring away part of it and adding water to the remainder until the suspension has a consistency of about 0.05 percent fibres. Partially fill a test-tube with the mixture.

NOTE — In the case of treated papers (like asphalt, tar, rubber and pyroxylin) and highly coloured papers, the chemical and the dye with which the paper has been treated shall be removed from the specimens by suitable methods before disintegrating the specimens.

18.2.4 Preparation of Slides — Thoroughly mix the test-tube suspension of the test pieces, insert the dropper to the middle of the suspension and withdraw a portion of the mixture. Place 0.5 ml of the suspension immediately on each end of the slide. (The test-tube shall be shaken and sample withdrawn separately each time). Evaporate a portion of the water, and carefully distribute the fibres evenly inside the 625 mm² area on the end of each slide with a dissection needle. Leave the slides on the hot plate until completely dried. Stain as described under 18.2.2 taking care that the unstained fibres on the slide are not touched by the fingers. Allow the slide to cool before adding the stain, otherwise confusing colours may be obtained.

18.2.5 Procedure — Examine the prepared slides microscopically, using a magnification of about 100 diameters. The identity of the fibres is given by the colour developed as shown below:

Wine Red — Linen and cotton.

Blue — Chemically prepared fibres low in lignin, from wood, straw, bagasse, bamboo and grasses.

Yellow — Fibres high in lignin, such as mechanical pulp, unbleached bast fibres and jute.

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